INTERLABORATORY STUDY 88-2B

VALIDATION OF A METHOD FOR RESIN AND FATTY ACIDS

AMPOULED STANDARDS IN TWO DIFFERENT SOLVENTS FOR DIRECT METHYLATION AND INSTRUMENTAL INJECTION

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Report Prepared by:
Laboratory Services Branch
Ontario Ministry of the Environment

JUNE 1990

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Table of Contents

1 OVERVIEW OF THE INTERLABORATORY STUDIES TO ASSESS THE METHOD FOR RESIN AND FATTY ACIDS 1

2 SUMMARY AND CONCLUSIONS 3

3 INTRODUCTION 4

4 PROCEDURE 4
- 4.1 Preparation of Ampoules 4
- 4.2 Ampoule Distribution 5
- 4.3 Analytical Methodology 5
- 4.4 Data Handling 5

5 RESULTS AND DISCUSSION 7

6 REFERENCES 11

7 APPENDIX 1 - LIST OF PARTICIPANTS AND CORRESPONDENCE 12

8 APPENDIX 2 16

1 OVERVIEW OF THE INTERLABORATORY STUDIES TO ASSESS THE METHOD FOR RESIN AND FATTY ACIDS

In 1987, an Analytical Working Group (AWG) was formed to develop an improved analytical procedure for the analysis of Resin and Fatty Acids (MISA Group 26 and PP3). The AWG consisted of representatives from the Ontario Forest Industries Association (OFIA), Ontario Ministry of the Environment (MOE), and Environment Canada (EC), under the MISA program.

Initial work was done during 1987 to assess the stability of standards, the pH adjustments to be made prior to extraction, column conditions, and other instrumental conditions. The results have been reported elsewhere (3). In May 1988, the Method for Resin and Fatty Acids, Draft 3 (4) was ready to be validated by means of an interlaboratory collaborative study. A series of four separate studies were undertaken to assess the new method.

Interlaboratory Study 88-2A

This was the first study initiated to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). Ampoules were distributed in June 1988. Four ampouled standards were provided to each participant, two to be spiked into reagent water and processed through the entire method, and two for direct methylation and injection into the analytical instrument.

The results indicated that the Draft Method produces similar results from all of the participants. Differences between laboratories may be attributed to differences in calibration of Dehydroabietic Acid. Some parameters are not recovered as well as others. This may be due to the difficulty of assuring the purity of the parameters.

The results from Interlaboratory Study 88-2A demonstrated that the Draft Method works, as long as the calibration of Dehydroabietic Acid is carried out in a precise and accurate manner. The initial review of the raw data raised concern among the AWG regarding the use of two different solvents to prepare the standard solutions. A small follow-up study was required (see Interlaboratory Study 88-2B below), using fresh stock solutions, to assess whether some of the differences in performance were introduced by the different solvents used to prepared the standards.

Interlaboratory Study 88-2B (This Study)

This was the second study to validate the performance of the Method for Resin and Fatty Acids, and was the follow-up to Interlaboratory Study 88-2A. Ampoules were distributed in August 1988. Four laboratories received two ampouled standards for direct methylation and instrumental injection. Each ampouled standard consisted of three parameters, one standard prepared in methanol and one standard prepared in methyl-t-butyl ether (MTBE). A third ampouled standard, consisting of the same three parameters dissolved in MTBE, but pre-methylated prior to sealing in the ampoules, was also provided to each laboratory. The third standard was to be used to verify that the four laboratories agreed in their calibration.

The results demonstrated that there was no significant difference between the mean of the results from standard prepared in methanol versus the mean of the results from the standard prepared in MTBE.

There was also no significant difference between the mean of the results from the standard prepared in MTBE that was not pre-methylated, versus the mean of the results from the standard prepared in MTBE that was pre-methylated.

Interlaboratory Study 88-3

This was the third interlaboratory study to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). A set of ten samples was distributed to the participants in November 1988. The sample sets consisted of a High Spike, Low Spike, and Blank of Reagent Water, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #1, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #2, and one unspiked Pulp and Paper Mill Effluent #3.

The results indicated that the laboratories were able to recover the spiked Resin and Fatty Acids from the reagent water samples. Results from the two different spiked pulp and paper mill effluents produced very variable results, with very few laboratories reporting results from the low spike effluent samples. Reasonable consistency of results between laboratories was achieved for the unspiked Pulp and Paper Mill Effluent #3.

The variability of the results suggested that there may have been problems recovering the spiked Resin and Fatty Acids from the effluent samples. It was decided to repeat this study in the spring of 1989. This study was reported as Interlaboratory Study 89-2 (see below).

Interlaboratory Study 89-2

This was the fourth (and final) interlaboratory study to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). A set of nine samples was distributed to the participants in March 1989. The sample sets consisted of a High Spike, Low Spike, and Blank in Reagent Water, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #1, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #2.

The results indicated that the laboratories were able to recover the spiked Resin and Fatty Acids from the reagent water samples. Results from the two different spiked pulp and paper mill effluents produced very variable results, with very few laboratories reporting results from Pulp and Paper Mill Effluent #2. The same sources of pulp and paper mill effluent were used for both Interlaboratory Study 88-3 and this study (89-2). After reviewing the mill processes, it was noted that Pulp and Paper Mill Effluent #2 was a biologically treated effluent. The absence of recovery of the spiked Resin and Fatty Acids from this effluent in both studies, suggests that the effluent was still biologically active and degraded the spiking material.

Separate reports have been prepared for each study.

2 SUMMARY AND CONCLUSIONS

Interlaboratory Study 88-2B was the follow-up study to Interlaboratory Study 88-2A, Resin and Fatty Acids, Ampoules for Spiking Reagent Water and Direct Methylation (4), conducted by the Quality Assurance Office, Laboratory Services Branch of the Ontario Ministry of the Environment. Study 88-2B was conducted to assess the possible difference in recovery of resin and fatty acids due to the solvent used to prepare the standard. One standard was to be prepared in methanol and the other standard was to be prepared in methyl-t-butyl ether (MTBE). To confirm that the instrumental calibration of the participants were comparable to each other, half of the standard prepared in MTBE was to be methylated prior to sealing in ampoules. This standard was to be directly injected into the instrument by each participant. The results would not be affected by each laboratory's own methylation efficiency and therefore should show comparable calibration.

Participants were provided with a set of three ampouled standards, two to be methylated and analyzed by direct instrumental injection, and one that had been methylated and was to be analyzed by direct instrumental injection. The results were to be reported as quickly as possible. This study involved the four laboratories which have members on the Analytical Working Group (AWG), a sub-committee of the Joint Technical Committee (JTC) for the MISA Pulp and Paper Sector. All results were reported after 9 weeks.

The results from this study demonstrated that the two different solvents used to prepared the resin and fatty acids standard did not affect the recovery of the parameters. The variability in the results from Interlaboratory Study 88-2A (4), and the differences in the results from the ampoules prepared in different solvents, therefore were due to the age of the ampoules prepared in methanol.

This study also demonstrated that two of the participants had very good agreement for their calibration. One of the other participants had low results and the fourth participant had high results. Both of the latter two laboratories demonstrated good within-laboratory precision, therefore it would not be difficult to adjust their calibration to agree with the first two laboratories.

The interlaboratory biases apparent in comparing within-lab repeatability vs. interlaboratory precision, significantly affects the ability to discern the significance of differences between the means for Ampoules A, B, and C. This also applies to discerning the significance of the changes in variability between laboratories.

3 INTRODUCTION

In June 1988, Interlaboratory Study 88-2A was conducted to assess the interlaboratory performance of the Method for Resin and Fatty Acids, Draft 3 (4), by the Quality Assurance Office, Laboratory Services Branch of the Ontario Ministry of the Environment. The results from this study have been reported elsewhere (5).

The ampouled standards used for Interlaboratory Study 88-2A were prepared at two different times, using two different solvents, methanol and methyl-t-butyl ether (MTBE). Based on the results of Interlaboratory Study 88-2A, it was decided to conduct a small study to determine if the solvent used to prepare the ampouled standards affected the recovery of the different parameters. Only the member laboratories of the Analytical Working Group (AWG), a sub-committee of the Joint Technical Committee (JTC) for the MISA Pulp and Paper Sector participated in this study.

A subset of the parameter list used in Interlaboratory Study 88-2A was chosen for this study. Fresh stock solutions were prepared, one using methanol and the other using MTBE as the solvent. Both solutions were prepared from the same lot number of neat chemical crystals and both were prepared at the same concentration. To determine if there were any differences in the instrumental calibration of participating laboratories, a portion of the stock solution prepared in MTBE was methylated by the QA Office. The methylated solution was provided to the participating laboratories as a third ampoule.

Three ampoules were provided to each participant. All analyses were to be done in duplicate. Details of ampoule preparation and distribution are given in Sections 4.1 and 4.2. Analytical methodology and data handling are presented in Sections 4.3. and 4.4. Final results are presented and discussed in Section 5.0.

4 PROCEDURE

4.1 Preparation of Ampoules

A 100 mg/mL solution, consisting of the compounds listed in Table 1, was prepared in methanol. The solution was subsequently chilled to -20 degrees Celcius prior to transferring to 5 mL amber ampoules and sealed with a flame.

A second 100 mg/mL solution was prepared in the same manner as above, using methyl-t-butyl ether (MTBE) as the solvent. This solution was divided into two 50 mL portions. The first portion was sealed into 5 mL amber ampoules. The second portion was methylated using the same procedure as described in Section 10.2 of the Draft Method (4). After the methylation step was completed, the second 50 mL portion of the standard in MTBE was sealed into 5 mL amber ampoules as above.

The standard solution prepared in methanol was labelled Ampoule A. The standard solution prepared in MTBE that was not methylated was labelled Ampoule B. The standard solution prepared in MTBE that was methylated was labelled Ampoule C.

TABLE 1 - Parameter List
Ampoules A, B, and C

Isopimaric Acid
Dehydroabietic Acid
Oleic Acid

4.2 Ampoule Distribution

A set of three (3) ampoules, one each of Ampoules A, B, and C, were packaged in a styrofoam shipping container. The packages were shipped via Purolator courier on August 12, 1988. Each package included a cover letter and instruction sheet. Copies are included in Appendix 1.

4.3 Analytical Methodology

All participating laboratories were required to use the Draft Method for Resin and Fatty Acids (4). The analytical principles of this method are described in Schedule 2 of the Draft Pulp and Paper Regulation (2). All participants were asked to provide details of any modifications they may have made to the method. Each participant was asked to analyze each ampoule in duplicate.

4.4 Data Handling

Results were submitted to the QA Office of Laboratory Services Branch, MOE, in written form by mail. All data were manually entered into an electronic spreadsheet.

Between-laboratory variability was determined by calculating the mean and standard deviation of the results reported. All results are presented in Section 5, Table 2.

Results were converted to percent recovery based on the design value of the ampoules. These values are also presented in Table 2.

To determine if there was a significant difference between the two solvents (methanol and MTBE), the Student's *t* value was calculated for each parameter, using the mean and standard deviations calculated for Ampoules A and B. To determine if there was a significant difference in the variability of results from the two different solvents, the F Test was calculated using the standard deviations calculated for each parameter in Ampoules A and B. The results for these calculations are presented in Section 5, Table 3.

Similarly, to determine if there was a significant difference in the variability of the results between the standard in MTBE methylated by the participant (Ampoule B) versus the standard methylated by the QA Office (Ampoule C), the F Test was calculated using the standard deviations calculated for each parameter from both ampoules. The results for these calculations are presented in Section 5, Table 4.

To graphically present the interlaboratory variability, the duplicate results from each laboratory were plotted for each ampoule . This graph is presented in Appendix 2.

5 RESULTS AND DISCUSSION

All of the participants were able to analyze the ampoules in duplicate.

To determine if there were analyst differences, the Pulp and Paper Research Institute had two different analysts perform the analysis in duplicate. These results are presented as P&P #1 and P&P #2 in Table 2. Analyst #1 demonstrated good within-laboratory precision. Analyst #2 demonstrated variable precision, most noticeably in the results from Ampoule B (MTBE). In a memo accompanying the results, R. Voss of the Pulp and Paper Research Institute noted that analyst #2 had consistently higher results for all three ampoules. He noted that this may be due to the higher Relative Response Factor that this analyst achieved for the Dehydroabietic Acid Calibration table (RRF for #1 = 1.04, RRF for #2 = 1.09). When compared to the mean results for all the laboratories, analyst #1 is consistently low, except for one result for Dehydroabietic Acid from Ampoule B (MTBE). Analyst #2 shows more variability, with some results from Ampoule A (methanol) higher than the mean, but results from the other ampoules were lower than the mean. There is insufficient data to determine if there is a significant difference in the variability of the performance of the two analysts.

The results from the Environmental Protection Service Nova Scotia (EPS-NS) demonstrate good within-laboratory precision from the duplicate results, except for Dehydroabietic Acid in Ampoule A (methanol). Their results for Ampoule A (methanol) were lower than the mean while their results from Ampoule B (MTBE) were higher than the mean.

The results from the Wastewater Technology Centre at the Canadian Centre for Inland Waters (CCIW) demonstrate good within-laboratory precision from the duplicate results. However their results are low compared to the mean from all the participants, suggesting that their calibration standards are more concentrated than the other participants.

The accompanying instruction sheet requested that the participants analyze a 25 µL aliquot of each standard. The Laboratory Services Branch of the Ontario Ministry of the Environment (MOE) routinely uses 50 µL aliquots of their standard. To confirm that the size of the aliquot did not affect the results, the MOE laboratory analyzed the ampoules using 25 µL and 50 µL aliquots, both in duplicate. The results in Table 2 indicate which aliquot was used. The duplicate results from Ampoules A and B (methanol and MTBE, respectively) show variability in the within-laboratory precision. However the results from Ampoule C (MTBE, methylated) are very precise for both aliquot sizes. This suggests that the methylation procedure used by the MOE introduces variability in the results. The aliquot size does not appear to affect the results.

TABLE 2 - ROUND ROBIN 88-2B: RESULTS FROM RESIN AND FATTY ACID AMPOULES

DISTRIBUTED: AUGUST 12, 1988

AMPOULE	PARAMETER	DESIGN (ug/mL)	P&P: #1 #1	P&P: #1 #2	P&P: #2 #1	P&P: #2 #2	EPS-NS #1	EPS-NS #2	CCIW #1	CCIW #2	MOE: 25 uL #1	MOE: 25 uL #2	MOE: 50 uL #1	MOE: 50 uL #2	MEAN	STD DEV	VAR
A (METHANOL)	OLEIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	ISOPIMARIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	DEHYDROABIETIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
B (MTBE)	OLEIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	ISOPIMARIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	DEHYDROABIETIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
C (MTBE: METHYLATED)	OLEIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	ISOPIMARIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	DEHYDROABIETIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

RESULTS EXPRESSED AS PERCENT RECOVERY OF THE DESIGN VALUE

AMPOULE	PARAMETER	P&P: #1 #1	P&P: #1 #2	P&P: #2 #1	P&P: #2 #2	EPS-NS #1	EPS-NS #2	CCIW #1	CCIW #2	MOE: 25 uL #1	MOE: 25 uL #2	MOE: 50 uL #1	MOE: 50 uL #2	MEAN	STD DEV
A (METHANOL)	OLEIC	48%	48%	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	ISOPIMARIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	DEHYDROABIETIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
B (MTBE)	OLEIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	ISOPIMARIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	DEHYDROABIETIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
C (MTBE: METHYLATED)	OLEIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	ISOPIMARIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	DEHYDROABIETIC	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

LEGEND: MTBE: METHYL T-BUTYL ETHER; #1: REPLICATE 1; #2: REPLICATE 2

P&P: PULP AND PAPER RESEARCH INSTITUTE
EPS-NS: ENVIRONMENTAL PROTECTION SERVICE, NOVA SCOTIA
CCIW: CANADA CENTRE FOR INLAND WATERS, WASTEWATER TECHNOLOGY CENTRE
MOE: ONTARIO MINISTRY OF THE ENVIRONMENT, LABORATORY SERVICES BRANCH

The MOE's results are high compared to the mean from all the participants. This appears to be a greater problem for the results from Ampoule A (methanol) compared to the results from Ampoules B and C (MTBE and MTBE, methylated, respectively). The MOE's calibration standards may be dilute compared to the other participants.

The results show that the Pulp and Paper Research Institute and the Environmental Protection Service, Nova Scotia have good agreement in calibration relative to the design values. The results from the Wastewater Technology Centre (CCIW) were low. The results from the Laboratory Services Branch, MOE were high. Both of the latter two laboratories did demonstrate good within-laboratory precision. Adjustment of the calibration standard of the CCIW and MOE laboratories would bring their results in agreement with those from the other two participants.

TABLE 3 - Statistical Calculations on Results in Table 2
Ampoule A (Methanol) vs. Ampoule B (MTBE)

	Oleic	Isopimaric	Dehydroabietic
$\overline{X}_{meth}, \overline{X}_{MTBE}$	618,616	1055, 1006	1011, 1006
s (total sample)	97.62	113.5	92.44
t	0.033	1.063	0.150
df	22	22	22
s_1^2, s_2^2	14758.3, 4302.9	19427.4, 6331.4	13174.1, 3917.3
$F = \frac{s_1^2}{s_2^2}$ (1=methanol) (2=MTBE)	3.43	3.07	3.36

For df=22, and a 95% Confidence Interval, t=1.717. For all three parameters, the calculated t value is less than 1.717. Therefore there is no significant difference between the standard prepared in methanol (Ampoule A) and the standard prepared in MTBE (Ampoule B).

For df_1=11,df_2=11, and a 95% Confidence Interval, F=2.82. For all three parameters, the calculated F value is greater than 2.82. Therefore there is a significant difference in the variability of the results reported from the standard prepared in methanol (Ampoule A) versus the standard prepared in MTBE (Ampoule B).

As indicated in Table 3, there was no significant difference in the mean value of the results from the two standards prepared in the different solvents. The results presented graphically in Figures 1 and 2 in Appendix 2 demonstrate the same pattern for all three parameters.

All the participants reported lower results for Oleic Acid from all of the ampoules. This may be due to a difference in the instrumental response factor for this parameter relative to Dehydroabietic Acid, the parameter used for the initial calibration as specified in the Draft Method (4). The lower results may also be due to the greater instability of this parameter or to lower purity of the standard, or both.

It was difficult to determine why the results from Ampoule A (methanol) were more variable than the results from Ampoule B (MTBE). Methanol is a less volatile solvent, therefore less evaporation should take place when sealing the solution into the ampoules. More variability should be introduced when sealing the solution prepared in MTBE into the ampoules.

TABLE 4 - Statistical Calculations on Results in Table 2
Ampoule B (MTBE) vs. Ampoule C (MTBE, Methylated)

	Oleic	Isopimaric	Dehydroabietic
s_2^2, s_3^3	4302.9, 13453.5	6331.4, 18573.3	3917.3, 11519.7
$F = \frac{s_2^2}{s_3^2}$	3.12	2.93	2.94

(2=MTBE)
(3=MTBE, Methylated)

For df_1=11,df_2=11, and a 95% Confidence Interval, F=2.82. For all three parameters, the calculated F value is greater than 2.82. Therefore there is a significant difference in the variability of the results reported from the standard prepared in MTBE and methylated by the participant (Ampoule B) versus the standard prepared in MTBE and pre-methylated by the QA Office (Ampoule C).

The results from Ampoule B (MTBE) show less variability than the results from Ampoule C (MTBE, Methylated). This difference is significant, as demonstrated in Table 4. This difference may be due to the increased volatility of the standard after it has been methylated. During the process of sealing the solution in the ampoules, the heat may have evaporated some of the solvent from each ampoule to a slightly different degree, contributing to the increased variability of the results from Ampoule C.

The results from this study demonstrated that the two different solvents used to prepared the resin and fatty acids standard did not affect the recovery of the parameters. The variability in the results from Interlaboratory Study 88-2A (5), and the differences in the results from the ampoules prepared in different solvents, is therefore attributed to the age of the ampoules prepared in methanol.

6 REFERENCES

1. Ontario Regulation 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act; Effluent Monitoring - General.

2. The Development Document for the Draft Effluent Monitoring Regulation for the Pulp and Paper Sector, March 1989; ISBN 0-7729-4764-3.

3. Minutes from the OFIA/MOE/EC Analytical Working Group, 1987 and 1988, inclusive. (Copies of all minutes were reported to the Joint Technical Committee for the Pulp and Paper Sector.)

4. Method for Resin and Fatty Acids; OFIA/MOE/EC Analytical Working Group; Draft, May 5, 1988.

5. Interlaboratory Study 88-2A; Validation of a Method for Resin and Fatty Acids; Ampoules for Spiking Reagent Water and Direct Methylation; February 1990, ISBN 0-7729-6749-0.

7 APPENDIX 1 - LIST OF PARTICIPANTS AND CORRESPONDENCE

List of Participants

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August 12, 1988.

TO: PARTICIPANTS OF RFA ROUND ROBIN (Part 2)

Please find enclosed three (3) 5 mL amber ampoules labelled Ampoule A, Ampoule B, and Ampoule C, and an instruction sheet. If you are missing any of the above items, please contact me at the above phone number immediately.

These ampoules are to be analyzed as soon as possible and the results forwarded to me as quickly as possible. This study is being conducted to compare the two solvents used in the ampoules in the first round robin (methanol and t-butyl ether), and also to compare the effect of pre-methylating the solution. All ampoules are for direct injection.

Please contact me if there are any questions.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist

ONTARIO MINISTRY OF THE ENVIRONMENT
RESIN AND FATTY ACID ROUND ROBIN (Part 2)
AUGUST, 1988.

AMPOULE PREPARATION INSTRUCTION SHEET

Upon receipt please ensure that your laboratory received three (3) 5 mL amber ampoules labeled Ampoule A, Ampoule B, and Ampoule C. Ensure that they are all intact. Store ampoules in a refrigerator at 4 degrees C until ready to process.

Do not open ampoules until ready to process.

ANALYZE ALL AMPOULES IN DUPLICATE.

Ampoule A and B

1. Break open ampoule carefully.

2. Add 25 µL of Ampoule A or B, 25 µL of the surrogate spiking solution (Section 6.10), 25 µL of the internal standard spiking solution (Section 6.7), 0.1 mL of methanol, and about 1.0 mL of diethyl ether to each of two concentration vessels.

3. Proceed with derivatization and analysis of the two duplicates as outlined in Sections 10.2 and 10.3.

4. Report the concentration of the spiking solution in Ampoules A and B in units of µg/mL (micrograms per millilitre).

Ampoule C

1. Break open ampoule carefully.

2. Add 25 µL of Ampoule C and 25 µL of the internal standard spiking solution (Section 6.7), to each of two concentration vessels.

3. Dilute to volume with the appropriate solvent and proceed with the analysis of the two duplicates as outlined in Section 10.3.

4. Report the concentration of the spiking solution in Ampoule C in units of µg/mL (micrograms per millilitre).

NOTE: Each ampoule contains only three compounds - Oleic, Isopimaric and Deydroabietic Acids.

8 APPENDIX 2

Figure 1 - Results From Ampoules A, B, and C

FIGURE 1: ROUND ROBIN 88-2B